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US006297353B1

(12) **United States Patent**
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(10) **Patent No.: US 6,297,353 B1**
(45) **Date of Patent: Oct. 2, 2001**

(54) **PROCESS FOR OBTAINING UNSAPONIFIABLE COMPOUNDS FROM BLACK-LIQUOR SOAPS, TALL OIL AND THEIR BY-PRODUCTS**

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/294,303**

(22) Filed: **Apr. 19, 1999**

(30) **Foreign Application Priority Data**

Apr. 22, 1998 (CL) 873/98

(51) **Int. Cl.**[7] **C08F 1/00**; C08F 1/04
(52) **U.S. Cl.** **530/205**; 530/230
(58) **Field of Search** 530/205, 230

(56) **References Cited**

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(57) **ABSTRACT**

A method for obtaining neutral compounds from black-liquor soaps, tall oil and tall oil by-products is disclosed. The method includes the steps of: (1) dehydrating black liquor soaps, neutralized tall oil, or neutralized tall oil by-products to form a dehydrated current; (2) distilling the dehydrated current in a distillation column; (3) collecting an essentially soap free distillate; and (4) collecting an essentially neutral compound free residue. Also disclosed is a method for obtaining a sterol concentrate from black-liquor soap solutions, neutralized tall oil, or neutralized tall oil by-products. In this method, a distillation operation is repeated until a current of sterols with the desired purity is obtained. The method yields a sterol concentrate with a level of purity over 96%. In another version of the method, a high purity sterol cocentrate is achieved by vacuum fractionation

17 Claims, 1 Drawing Sheet

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Hewlett Packard chromatographer, model HP 5890, series 2, capillary column HP-5, 30 m long, 0.32 mm diameter, 0.25 mm film	
Detector temperature	320° C.
Carrier flux (He)	0.92 ml/min
Split	60:1
Program	15 min
Injection	0.5 μl

b) Sample Preparation

Accurately weigh 0.1 mgr., 100 mgr. of sample
Dissolve in 25 ml of tetrahydrofurane (THF)
Add 500 μl of this solution in a silanization vial
At the same time, precisely weigh 0.1 mgr., 50 mgr. of 5β-colestan-3α-ol
Dissolve in 100 ml of n-propanol
Add in the tube 500 μl of the 5 β-colestan-3α-ol solution
Dry under nitrogen atmosphere
Add 300 μl of Bis (trimethylsilyl) trifluoroacetamide (BSTFA)
Add 300 μl of pyridine
Maintain the solution at 70° C. for 10 minutes
Dry under inert atmosphere
Dissolve with 500 μl of THF
Note: The reagents must have an analytic grade.

c) Calculations

Record the area of the compound of interest
Record the area of 5β-colestan-3 α-ol
Calculate the weight percentage of the compound of interest through the following formula:

$$\% X = \frac{A_x \cdot M_p}{A_p \cdot M_m} \cdot 100$$

Where,

X: percentage in weight of the compound of interest

A_x: chromatographic area of the compound of interest

M_p: pattern added mass (5β-colestan-3α-ol)

A_p: pattern chromatographic area (5β-colestan-3 α-ol)

M_m: sample added mass

Although the invention has been described in considerable detail with reference to certain preferred versions, one skilled in the art will appreciate that the present invention can be practiced by other than the preferred versions, which have been presented for the purpose of illustration and not of limitation. Therefore, the spirit and scope of the appended claims should not be limited to the description of the preferred versions provided herein.

What is claimed is:

1. A method for obtaining neutral compounds of tall oil pitch comprising the following steps:

neutralizing tall oil pitch with an aqueous solution of sodium hydroxide, potassium hydroxide or a mixture thereof to form a neutralized solution;

dehydrating the neutralized solution to form a dehydrated current;

feeding the dehydrated current to a distillation column selected from the group consisting of molecular distillation columns and short path distillation columns;

distilling the dehydrated current in the distillation column;

collecting a distillate current including the neutral compounds from the distillation column; and

collecting a residue current from the distillation column.

2. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

feeding the neutralized solution to a spray dryer;

contacting the neutralized solution to a hot gas current at temperatures between 150 and 250° C.; and

collecting the dehydrated current.

3. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

feeding the neutralized solution to a falling film evaporator; and

collecting the dehydrated current,

wherein the temperature of an evaporation surface of the evaporator is between 100 and 250° C. and the pressure of the evaporator is between 100 and 1000 mbar.

4. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

centrifuging the neutralized solution;

collecting a light phase resulting from the centrifuging;

feeding the light phase to a spray dryer wherein the light phase contacts a hot gas current at temperatures between 150 and 250° C.; and

collecting the dehydrated current.

5. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

centrifuging the neutralized solution;

collecting a light phase resulting from centrifuging;

feeding the light phase to a falling film evaporator, wherein the temperature of an evaporation surface of the evaporator is between 100 and 250° C. and the pressure of the evaporator is between 100 and 1000 mbar; and

collecting the dehydrated current.

6. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

mixing the neutralized solution with one or more unsaponifiable fractions of black liquor soaps to form a mixture;

centrifuging the mixture;

collecting a light phase of the centrifugation;

feeding the light phase to a falling film evaporator, wherein the temperature of an evaporation surface of the evaporator is between 100 and 250° C. and the pressure of the evaporator is between 100 and 1000 mbar; and

collecting the dehydrated current.

7. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

centrifuging the neutralized solution;

collecting a light phase of the centrifugation;

mixing the light phase with one or more unsaponifiable fractions of black liquor soaps;

feeding the light phase to a falling film evaporator, wherein the temperature of an evaporation surface of the evaporator is between 100 and 250° C. and the pressure of the evaporator is between 100 and 1000 mbar; and

collecting the dehydrated current.

8. The method of claim 1 characterized in that the step of dehydrating the neutralized solution to form the dehydrated current comprises:

mixing the neutralized solution with one or more unsaponifiable fractions of black liquor soaps to form a mixture;

feeding the mixture to a falling film evaporator, wherein the temperature of an evaporation surface of the evaporator is between 100 and 250° C. and the pressure of the evaporator is between 100 and 1000 mbar; and

collecting the dehydrated current.

9. The method of claim 1 further including the steps of:

mixing the distillate current with an aqueous solution of an alkali hydroxide to form a distillate solution;

heating the distillate solution at a temperature between 100 and 300° C. for at least 15 minutes;

separating a non-aqueous phase from the distillate solution; and

collecting the non-aqueous phase.

10. The method of claim 9 further including the step of:

feeding the non-aqueous phase to a distillation column selected from the group consisting of molecular distillation columns and short path distillation columns,

wherein the distillation column includes a vertical surface of heated evaporation, a revolving scraper and an inner condenser located at less than 100 centimeters from the vertical surface wherein

the non-aqueous phase is fed on the vertical surface and spread on the vertical surface as a thin layer while it is heated,

a second distillate current is collected from the inner condenser,

a second residue current is collected from a bottom portion of the distillation column,

the vertical surface is heated at temperatures between 50 and 200° C. and

the pressure is not higher than approximately 0.5 mbar.

11. The method of claim 1 further including the steps of:

feeding the distillate current to a fractionation column having a reboiler and a condenser, the reboiler and the condenser operating at a pressure less than 1 mbar; and

collecting one or more secondary distillate currents.

12. The method of claim 1 characterized in that:

the distillation column includes a vertical surface of heated evaporation, a revolving scraper and an inner condenser located less than 100 centimeters from the vertical surface wherein

the dehydrated current is fed on the vertical surface and spread on the vertical surface as a thin layer while the dehydrated current is heated,

the distillate current is collected from the inner condenser,

the residue current is collected from a bottom portion of the distillation column,

the vertical surface is heated at temperatures between 150 and 400° C., and

the pressure is not higher than approximately 0.5 mbar.

13. The method of claim 12 further including the steps of:

feeding the distillate current to a distillation column selected from the group consisting of molecular distillation columns and short path distillation columns, the distillation column including a vertical surface of heated evaporation, a revolving scraper and an inner condenser located less than 100 centimeters from the vertical surface wherein

the distillate current is fed on the vertical surface and spread on the vertical surface as a thin layer while the distillate current is heated,

a second distillate current is collected from the inner condenser,

a second residue current is collected from a bottom portion of the distillation column,

the vertical surface is heated at temperatures between 50 and 250° C., and

the pressure is not higher than approximately 0.5 mbar.

14. The method of claim 13 further including the steps of:

feeding the second distillate current to a distillation column selected from the group consisting of molecular distillation columns and short path distillation columns, the distillation column including a vertical surface of heated evaporation, a revolving scraper and an inner condenser located less than 100 centimeters from the vertical surface wherein

the second distillate current is fed on the vertical surface and spread on the vertical surface as a thin layer while the second distillate current is heated,

a third distillate current is collected from the inner condenser,

a third residue current is collected from a bottom portion of the distillation column,

the vertical surface is heated at temperatures between 100 and 250° C., and

the pressure is not higher than approximately 0.5 mbar.

15. The method of claim 14 wherein the third distillate current contains not less than 60% of sterols.

16. The method of claim 14 further including the steps of:

feeding the third distillate current to a distillation column selected from the group consisting of molecular distillation columns and short path distillation columns, the distillation column including a vertical surface of heated evaporation, a revolving scraper and an inner condenser located less than 100 centimeters from the vertical surface wherein

the third distillate current is fed on the vertical surface and spread on the vertical surface as a thin layer while the third distillate current is heated,

a fourth distillate current is collected from the inner condenser,

a fourth residue current is collected from a bottom portion of the distillation column,

the vertical surface is heated at temperatures between 50 and 200° C., and

the pressure is not higher than approximately 0.5 mbar.

17. The method of claim 16 wherein the fourth distillate current contains not less than 80% of sterols.

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